National Aeronautics and Space Administration

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BRINGING NASA TECHNOLOGY DOWN TO EARTH

NASA’s Healing and Strengthening of Commercial SiC Tows Using Microwave Irradiation

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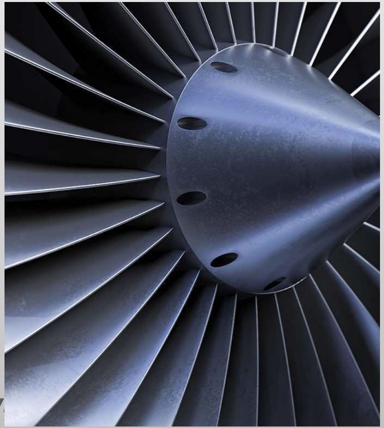
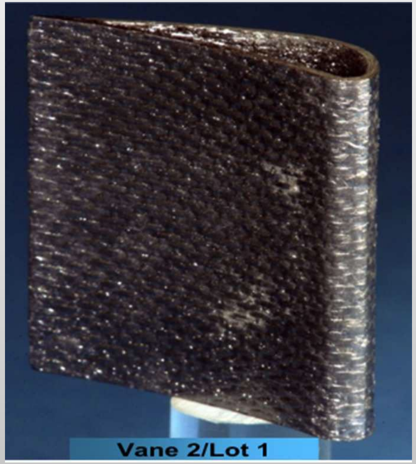
Background and Motivation

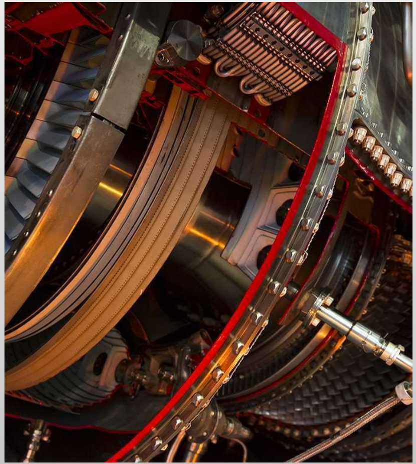
- Fiber Reinforced Ceramic Matrix Composites (CMC)
  - Silicon Carbide Matrix/Silicon Carbide Fiber (SiC/SiC)
- Applications: CMC Turbine blades and heat exchanger panels for space launch vehicles
  - SiC/SiC CMCs are enabling materials for a variety of advanced applications where lightweight reusable structural materials are required to operate for long time periods within extreme high-temperature environments, such as the hot-section components of military and commercial gas turbine engines.



Background and Motivation

- **Benefits: SiC/SiC offer higher structural reliability and temperature capability.**
  - Compared to superalloy metallic components, there is a reduction in engine weight, cooling air requirements, fuel burn, harmful exhaust emissions as well as improved thrust-to-weight ratio.
- **Goal: Develop an Ultra High Temperature (UHT) SiC Fiber using commercially available SiC fibers.**





Background and Motivation

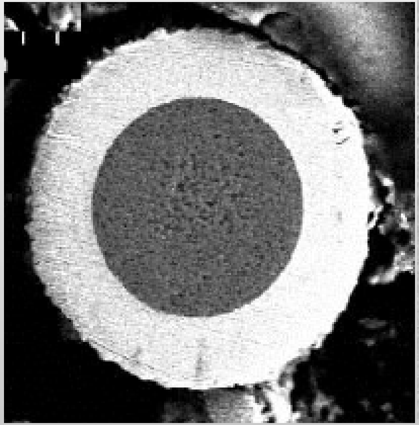
Experimental work

- Convert lower strength commercially available SiC fiber to high strength SiC fiber using 3 heat treatments steps.

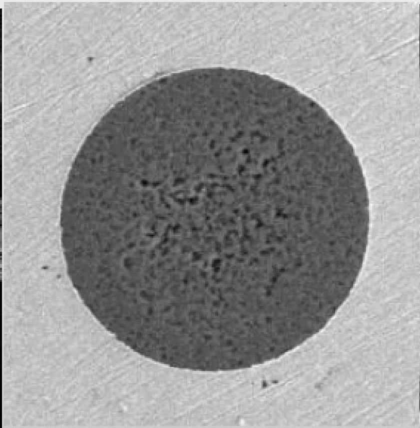
Results

- Lox-M SiC Fibers with 3 heating treatments.

Too many processing steps in high temperature furnace resulting in microstructure degradation.



LM/14D/14B/18S



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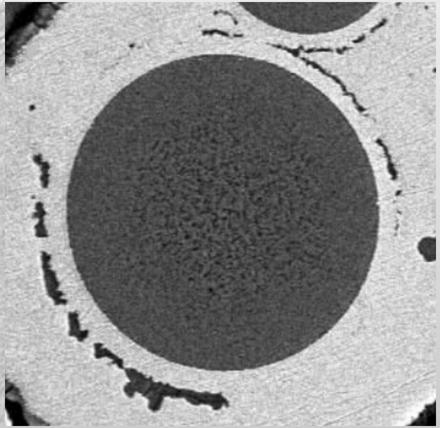

LM/14D/16B/18S

Need New Approach

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- Susceptors are materials that can absorb electromagnetic energy and convert the energy into heat.
- SiC is used as a susceptor in microwave sintering.
- Aha moment! Use a microwave (MW) furnace to develop high strength SiC tows from commercial sources.

- Single Mode Microwave Furnace

NASA GRC's MW capability is a single mode 2.45 GHZ MW furnace with up to 2 KW of power. During operation, a standing wave is obtained. Sintering can be performed in either the electric field (E-Field) or magnetic field (M-Field).

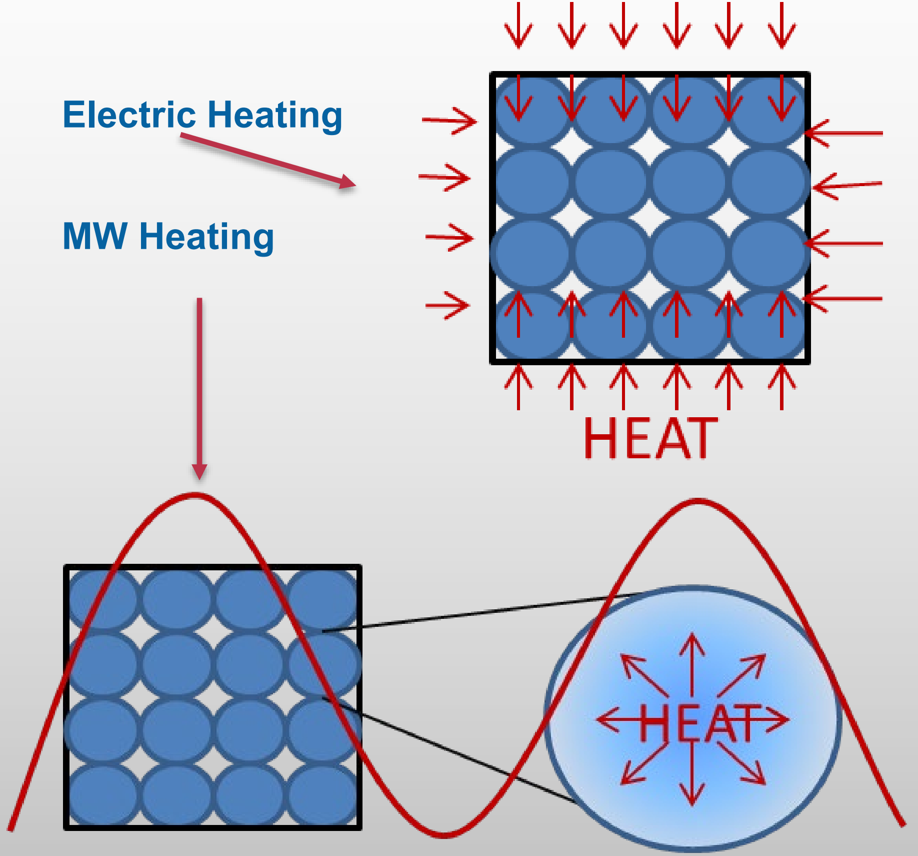
Electric Furnace vs Microwave Furnace for SiC Tows

Electric Furnace

- Conduction/convection heating
- Heating rates of 4 ^{o}C/min
- SiC surfaces exposed to long periods of gradient temperatures during heating and cooling resulting in non uniform microstructures.
- Requires high power, high energy.

Microwave Furnace

- MW coupling with a susceptor induces electron excitation resulting in heat generation.
- Fast heating rates up to 1000 ^{o}C/min.
- Lower processing temperatures and power needs.
- Uniform heating and cooling with high density microstructure.
- Sinter in either E or M-field *single mode furnace.*



Promising Results: Microstructure SEM

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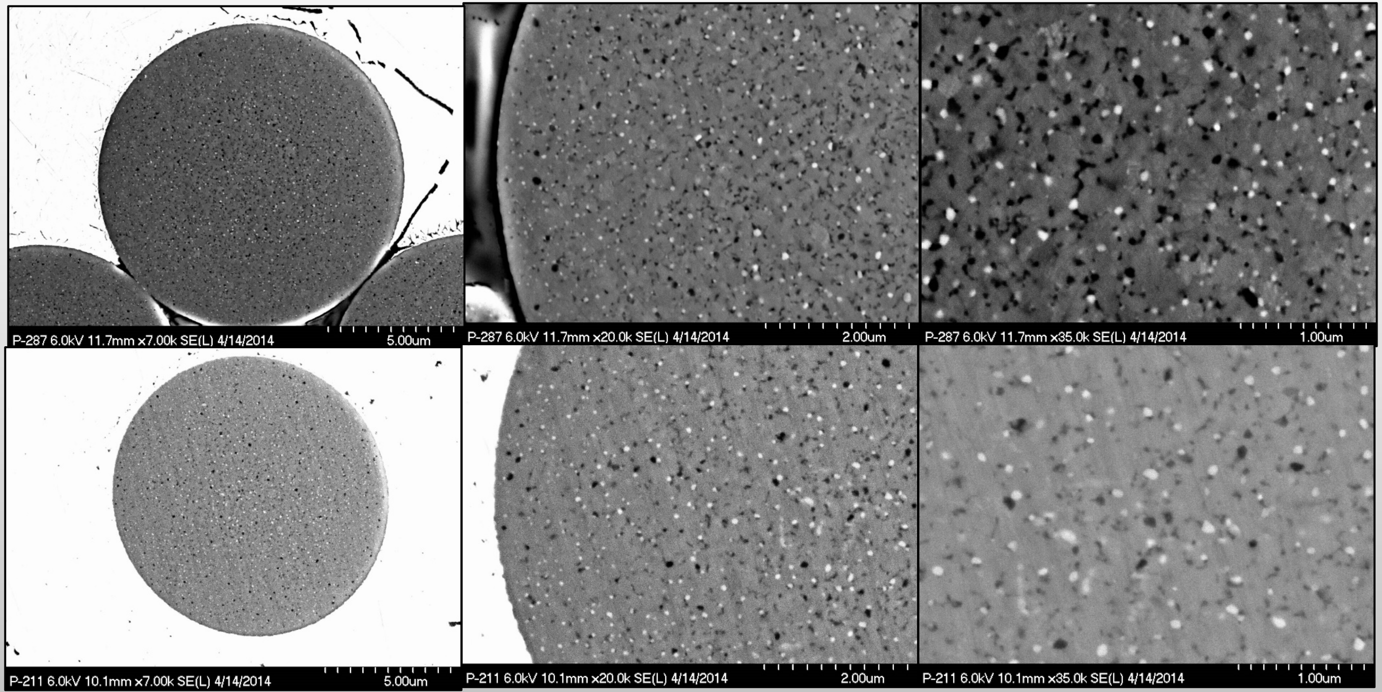
- **SEM of Microwave heated Sylramic SiC tow showed changes in microstructure porosity.**

- **As Receive Sylramic SiC Fiber**

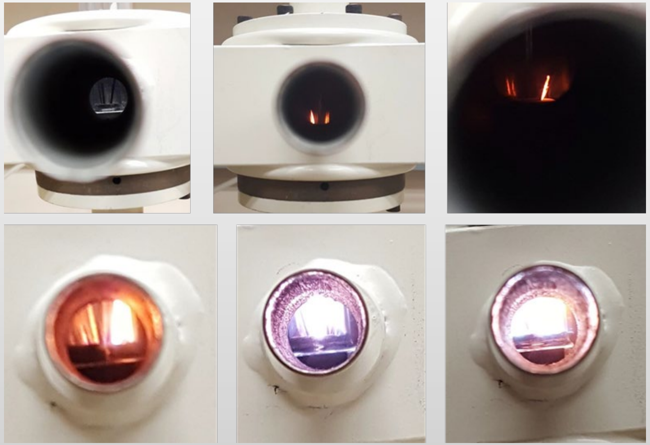
- **Microwave treated Sylramic SiC Fiber**



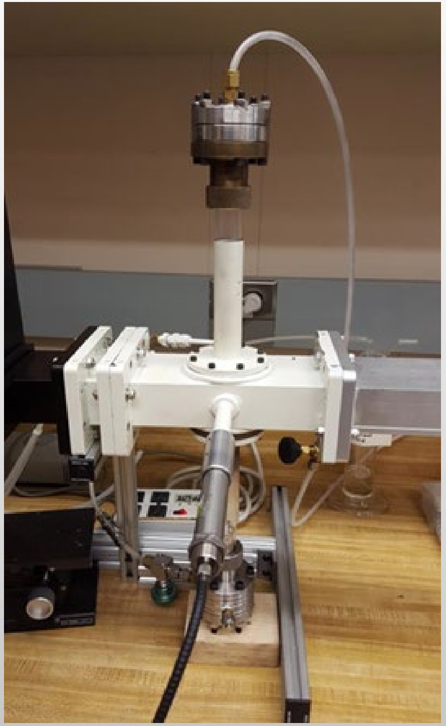
Microwave Processing Challenges

- **MW processing can be difficult.**
- **Many Processing Parameters**
  - Calibration/Standing wave tuning
  - Power levels and ramp rate
  - Temperature limits
  - Quartz tube preparation
  - Sintering area is small
  - Sample position
  - Gas atmosphere
- **Inert gases can cause plasma generation.**
- **Power levels, power ramp rates and sample position play a role in coupling, temperature, arcing events and/ or plasma generation.**
- **Surface charge buildup can lead to arcing events.**
- **Once setup is routine, processing becomes easy and fast.**

Beginning of coupling



Plasma Generation



MW Processing and Tensile Test

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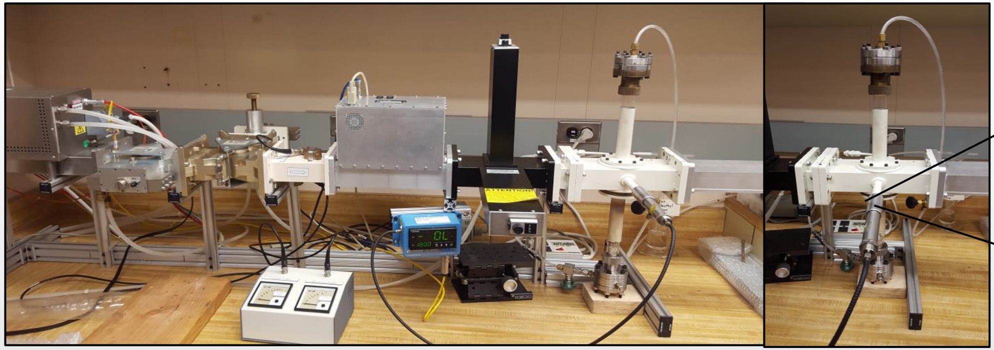
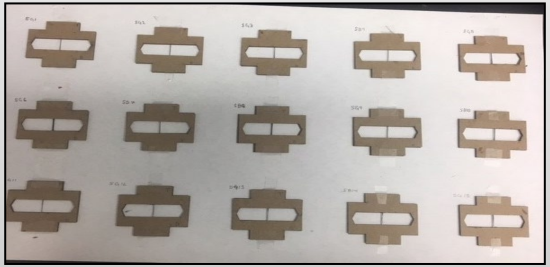
- **Prepare samples for loading in MW furnace**
- **Heat treat sample**
- **Furnace cools for 30 to 60 minutes**
- **Samples are prepared for tensile testing then tested**
- **Samples can be sintered and tested in 1 day with time to spare.**



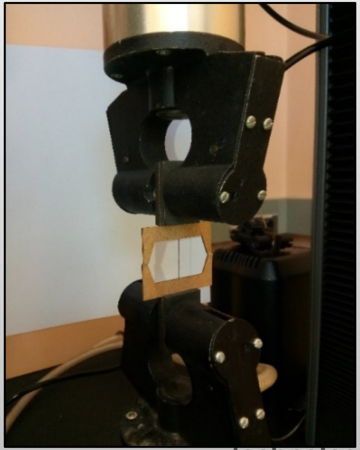
2.45 GHz Single Mode Microwave Furnace

Coupled Sample

1. Heat Treating Sample



2. Heat Treated Samples Prepared for Tensile Testing



3. Sample Loaded in Tensile Test Rig

First Experiments with Sylramic Tows

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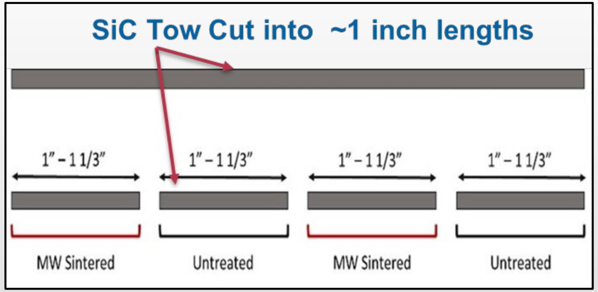
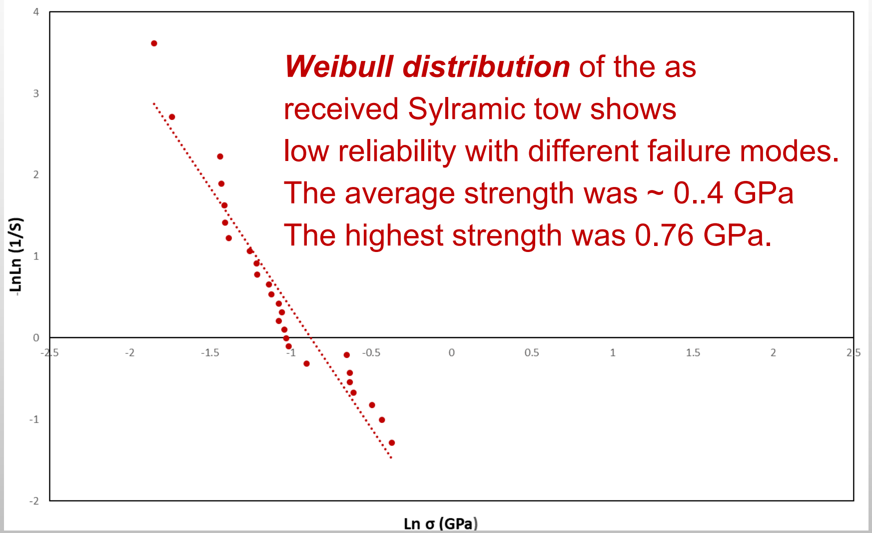


- **Electric (E) Field MW Sintering**
  - Samples must couple with field to sinter.

Sample Name	Gas Environment	Annealing time (mins)	P_{max} (W)	T_{max} (°C)	MW coupling (Y)es/(N)o	Arc (Y)es/(N)o	Strength σ GPa
3.1.1	Ar	2	300	N/A	N	N	0.27
3.2	Ar	3	300	N/A	N	N	0.34
3.3	Ar	5	300	N/A	N	N	0.06
3.5	Ar	2.5	300	N/A	N	N	0.27
3.6	**Ar**	**4**	**300**	**1350**	**Y**	**N**	**0.62/0.42**
3.7	**Ar**	**2**	**300**	**1400**	**Y**	**N**	**0.04/.14**
4.1	Ar	2	400	N/A	N	Y	0.4/.073
4.2	**Ar**	**4**	**200**	**1200**	**Y**	**N**	**0.18/0.15**
4.3	**Ar**	**3**	**400**	**1500**	**Y**	**N**	**0.23/0.41**
4.4	**Ar**	**1.3**	**100**	**1600**	**Y**	**N**	**1.16/1.09**
SiC B/N 3	**N**	**2**	**400**	**1750**	**Y**	**Y**	**0.95**



Sample Name	Highest Strength/ Sintering Temperature	% Increae to Sylramic Average Strength 0.4 GPa	% Increae to Sylramic Highest Strength 0.76 GPa
4.4 tow 1	1.16 GPa/ 1600 °C	190%	52.63%
4.4 tow 2	1.09 GPa/ 1600 °C	172.5%	43.42%
SiC-B/N-3 (1 tow)	0.95 GPa/1750 °C	138%	25%

MW Coupled Samples heated at ≥1600 °C show restorative strengthening of SiC tow strength.

Experimental Results Lead to More Questions!

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- Can we repeat the results?
- Can the strength of the SiC tows be restored to Vendor strength?
- The electric field was used to heat treat the SiC tows. Will the strength change in the magnetic field?
- SiC tows are manufactured with different compositions. Do the non-SiC constituents effect MW processing parameters?
- Will the strength go up or down in a high strength tow?
- What is the lowest temperature we can go?
- What is the lowest power we can use?

Let’s take a look!

Strength of SiC Tows Under Examination

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- **Chemical Composition and Tensile Strength of as received SiC tows.**

Fiber Trade Name	Vendor	Non-SiC Elements wt. %	Vendor Tensile Strength σ (GPa)	NASA Measured Avg. Strength σ (GPa)/ High Value
Hi-Nicalon S (NOX grade)	**Nippon Carbon**	0.7 Ox	2.8	2.89/3.35
Sylramic	**ATK-COI Ceramics**	0.1 Ox, 1.2 B, 2.4 Ti	3.2	0.398/0.775
Tyrano Lox-M	**UBE Industries**	10 Ox, 2 Ti	3.3	0.389/0.599

Tensile Testing Results Sylramic SiC Tows

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E-Field

- 203 % increase in average tensile strength.
- Temp. ranges: 1200 ˚ C to 1600 ˚ C .
- Power ranges: 60 to 100 Watts.
- Dwell times: 5 to 15 minutes.
- Longer dwell times at higher temperatures ≥ 1400 ˚ C , could show complete healing to full strength or higher.

M-Field

- 363 % increase in average tensile strength.
- **Temp. ranges: < 800** ˚ C **.**
- Power ranges: 60 to 100 Watts.
- Dwell times: 5 to 15 minutes.
- Sustained arcing at 1 minute showed promise.

Summary

- Vendor strength or higher may be possible in M-Field.
- E-Field sintering limited by non-SiC (TiB_2) in Sylramic. Coupling power will determine temperature/dwell time in both fields.

E or M Field SiC Tow Type	**Temp (°C) / Power (W)**	**Time (mins)**	**$\sigma_{AVG}/\sigma_{High}$ (GPa)**	**Avg St. Dev. +/-**
Electric Field	1000 C/ 60 W	5	0.71	0.14
Sylramic	1000 C/ 56 W	20	0.32	0.01
Avg Stemgth	1200 C/ 40 W	15	0.98	0.06
before MW	***1400 C/ 64 W***	***5***	***1.21***	***0.03***
0.4 GPa	1600 C/ 100 W	1.3	1.13	0.04
0.71 GPa				
Manf. σ				
3.2 GPa				
Magnetic Field	<800 C/50 W	20	0.94	0.16
SYlramic	<800 C/60 W	5	1.41	0.23
	<800 C/60 W	***15***	***1.85/3.04***	***0.77***
	<800 C/ 120 W	***10***	***1.5/2.11***	***0.37***
	<800/ 100 W	1 (arc)	1.42	0.25

Tensile Testing Result Hi-Nicalon-S

E-Field

- 14.87 % increase in Vendor tensile strength.
- Temp. ranges: 1000 ˚ C to 1200 ˚ C .
- Power ranges: 50 to 60 Watt.
- Dwell times: 0.3 to 10 minutes.
- Longer dwell times with higher power decreased strength.

M-Field

- 11.42 % increase in vendor strength.
- Temp. ranges: 1000 ˚ C to 1400 ˚ C .
- Power ranges: 50 to 100 Watt.
- Dwell times: 0.3 to 30 minutes.

Summary

- Tow strengthening in both fields.
- Coupling power will determine temperature and dwell time in both fields.
- As temperature and power rises dwell time decreases

E or M Field SiC Tow Type	Temp (^{o}C) / Power (W)	Time (mins)	$\sigma_{AVG}/\sigma_{High}$ (GPa)	Avg St. Dev. +/-
Electric Field	***1000 C/ 60 w***	***0.3***	***3.02/3.22***	***0.24***
Hi-Nicalon S	***1000 C/ 60 w***	***5***	***3.32/3.63***	***0.31***
	1000 C/ 60 w	***10***	***3.04/3.21***	***0.11***
Avg Stemgth	1000 C/ 80 w	25	1.00	0.23
before MW	1000 C/ 90 w	15	2.10	0.21
2.89 GPa	1000 C/ 140 w	20	1.26	0.05
Manf.σ 2.89 GPa	***1200 C/ 52 W***	***5***	***3.00/3.27***	***0.23***
Magnetic Field	900 C/ 100W	10	2.40	0.31
Hi-Nicalon S	1000 C/ 100 W	2	3.04	0.37
	1000 C/ 70 W	15	3.04	0.20
	1000 c/ 50 W	30	3.10	0.10
	1030 C/ 50 W	20	3.10	0.30
	1040 C/ 50 W	***10***	***3.10***	***0.06***
	1060 C/ 60 W	***5***	***3.22/3.55***	***0.21***
	1200 C/ 60 W	5	3.10	0.40
	1340 C/100 W	***5***	***3.20/3.67***	***0.16***
	1440 C/ 70 W	5	3.01	0.17

Tensile Testing Results Tyrano Lox-M

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- **M- Field**
  - 173.68 % increase in avg. strength.
  - Temp. ranges: 1000 ˚ C to 1400 ˚ C .
  - Power ranges: 50 to 80 Watts.
  - Dwell times: 10 to 20 minutes.
- **Summary**
  - Relatively good results for M-Field healing, more studies needed.
  - Tyrano Lox-M no longer available.
  - Studies on other Tyrano tows can be considered.

E or M Field SiCTow Type	Temp (°C) / Power (W)	Time (mins)	$\sigma_{AVG}/\sigma_{High}$ (GPa)	Avg St.Dev. +/-
M-Field Tyrano Lox-M ***Avg Stemgth before MW 0.38 GPa High 0.59 GPa Manf. σ 3.3 GPa***	***1000 C/ 50 w***	***20***	***1.04/1.34***	***0.25***
	1000 C/ 60 W	5	0.76	0.12
	1000 C/ 60 W	15	0.68	0.20
	1000 C/ 70 W	15	0.57/0.71	0.33
	900 C/ 80 W	5	0.62	0.55
	1000 C/ 80 W	***10***	***0.87/1.40***	***0.32***

Did We Answer All the Questions?

- Can we repeat the results? **YES!**
- Can the strength of the SiC tows be restored to manufactured strength? **YES !**
- The electric field was used to heat treat the SiC tows. Will the strength change in the magnetic field? **YES!**
- SiC tows are manufactured with different compositions. Do the non-SiC constituents effect MW processing parameters? **YES, Sintering/healing parameters depend on field, non-SiC constituents and coupling power.**
- Will the strength go up or down in a high strength tow? **Both!**
- What is the lowest temperature we can go? **< 800 ˚ C (M-Field) up to 1000 ˚ C depending on non-SiC constituents and coupling power.**
- What is the useful power we can use? **~ 60 W.**

Single Mode MW Sintering Summary

- MW sintering can restore low strength commercially available SiC tows to manufactured strengths.
- MW sintering can increase the strength of commercially available SiC tows.
- Single Mode MW sintering can heal damaged SiC tows.
- SiC tows strengths can be tailored to application need.

Benefits

- Fast Process
- Low Energy Cost Effective Processing
- Healing Capabilities

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Questions?

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Thank you!